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PATENT

Trimethylolalkanes and process for preparing them

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The object of the present invention is the preparation of trimethylolalkanes and, in particular, of trimethylolpropane.

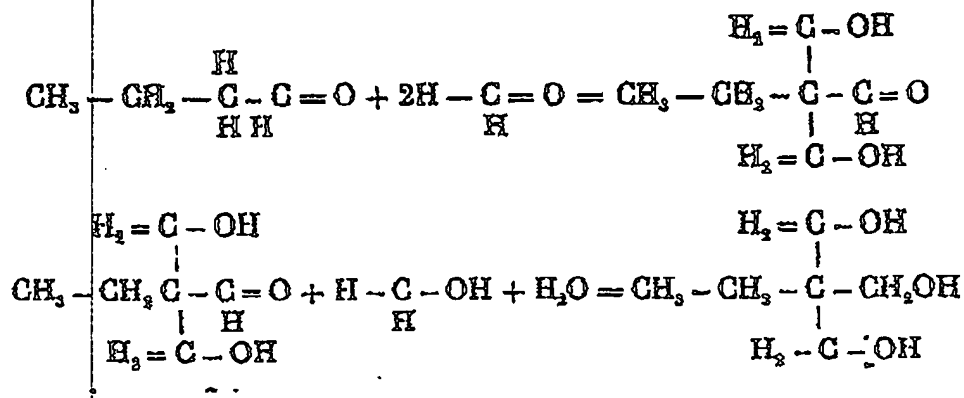
It has long been known that pentaerythritol can be prepared by condensing formaldehyde with acetaldehyde in a basic medium.

The present invention consists, for preparing the substances indicated above, in performing similar reactions but starting from aldehydes other than acetaldehyde, so as to prepare compounds containing three methylol groups attached to the same carbon, the fourth valency of the carbon being satisfied by a radical originating from a saturated hydrocarbon.

In particular, using normal-butyric aldehyde as base aldehyde, rather than acetaldehyde, the product, in accordance with the invention, is trimethylolpropane.

In this case, the two hydrogen atoms borne by the carbon adjacent to the functional group each react with one molecule of formaldehyde to give two methylol radicals, while the aldehyde group undergoes combination with a third molecule of formaldehyde to form another methylol group, in accordance with the conventional mechanism of a Cannizzaro reaction. At the same time formic acid is released, and is immediately neutralized by the base in whose presence the reaction is conducted.

Overall, the conversions may be written schematically as follows:



In practice it will be possible to operate by reacting one molecule of normal-butyric aldehyde with three molecules of formaldehyde in the presence of lime at a temperature in the region of 60°C and under atmospheric pressure.

The yields are in the region of 66%. To make, for example, 100 kg of trimethylolpropane in the course of a certain operation involves consumption of the following:
101.7 kg of formaldehyde and
81.3 kg of butyric aldehyde.

Following purification, the trimethylolpropane is in the form of a relatively hard solid which has the appearance of a greyish white technical-grade paraffin.

Its melting point is 53°C and it distils at 194°C under 2 mm pressure. This substance very rapidly absorbs atmospheric water vapour and undergoes superficial liquefaction on contact with the air.

By vacuum distillation it is relatively easy to separate trimethylolpropane from its dimer, which is analogous to dipentaerythritol; the operation is more convenient than for pentaerythritol since, in the course of this distillation, or more precisely this

concentration, the higher condensation products, analogous to sugars, are completely destroyed by carbonization.

As a consequence it is possible with ease to vary the industrial quality of trimethylolpropanes, since varying the quantity of heat supplied for the vacuum concentration automatically involves purification to a greater or lesser extent.

The purity of trimethylolpropane can be assessed with a fair degree of exactitude in the course of a nitration test. With a pure product, the nitrate has a melting point of 51.6°, a density of 1.48, and the yield reaches 91.5%.

A less pure product gives, in addition, a certain amount of nitrate derivative of the dimer, in the form of an oily liquid, having the appearance of nitro-glycerine.

It will of course be possible to make numerous modifications without departing from the scope of the invention; in particular, it will be possible to replace the butyric aldehyde by other homologues of acetaldehyde.

CLAIMS

1. Process for preparing trimethylolalkanes, and especially trimethylolpropane, which consists in reacting formaldehyde with a higher homologue of acetaldehyde and, in particular, normal-butyric aldehyde.

2. Specific embodiments of this process which are applicable to the preparation of different technical grades of trimethylol, in which a greater or lesser degree of purity is determined by the manner in which the final concentration is conducted, the said final concentration being accompanied by destruction of the sugars to an extent which increases in proportion with the duration of the carbonization.